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[Title of the Invention] METHOD FOR WASHING HOLLOW FIBER  
MEMBRANE MODULE AND FILTRATION DEVICE USED FOR THE METHOD

[Abstract] (amended)

[Problem to be Solved]

This invention provides a method for washing a hollow fiber membrane module that allows a long period of continuous stable filtration operation and a filtration device used for the method.

[Solution]

The method includes: performing a pressurizing process of introducing gas having pressure lower than pressure of gas discharged from a raw liquid side of a hollow fiber membrane, from a filtrate side of the hollow fiber membrane, with the raw liquid side of the hollow fiber membrane being filled with liquid, and washing the raw liquid side of the hollow fiber membrane with bubbles during or after the pressurizing process. The filtration device used for the washing method includes: a hollow fiber membrane module 1 having gas introduction ports 6 and 9 on the filtrate side and the raw liquid side, respectively; a liquid supply pump that supplies raw liquid to the hollow fiber membrane module 1; a pressurized gas supply device that supplies pressurized gas to the hollow fiber membrane module; and a control device that operates the liquid supply pump and the pressurized gas supply device.

[Claims for the Patent]

[Claim 1]

A method for washing a hollow fiber membrane module comprising:

performing a pressurizing process of introducing gas having pressure lower than pressure of gas discharged from a raw liquid side of a hollow fiber membrane, from a filtrate side of the hollow fiber membrane, with the raw liquid side of the hollow fiber membrane being filled with liquid; and

washing the raw liquid side of the hollow fiber membrane with bubbles during or after the pressurizing process.

[Claim 2]

The method for washing a hollow fiber membrane module according to claim 1, wherein said method is of an external filtration system in which raw liquid is supplied from an outer surface side of the hollow fiber membrane, and filtrate is taken out from an inner surface side of the hollow fiber membrane.

[Claim 3]

The method for washing a hollow fiber membrane module according to claim 1 or 2, wherein the pressure of gas introduced in the pressurizing process is 1.0 to 5.0 kg/cm<sup>2</sup>.

[Claim 4]

A filtration device comprising:

a hollow fiber membrane module having gas introduction ports on a filtrate side and a raw liquid side, respectively;

a liquid supply pump that supplies raw liquid to said hollow fiber membrane module;

a pressurized gas supply device that supplies pressurized gas to said hollow fiber membrane module; and

a control device that operates said liquid supply pump and said pressurized gas supply device,

wherein said control device is configured to perform a pressurizing process of supplying gas having pressure lower than pressure of gas discharged from the raw liquid side of the hollow fiber membrane, from the gas introduction port on the filtrate side to the hollow fiber membrane module, with the raw liquid side of the hollow fiber membrane being filled with the raw liquid, after the supply of the raw liquid to the hollow fiber membrane module is stopped, and perform a bubble washing process of introducing gas from the gas introduction port on the raw liquid side of the hollow fiber membrane to the hollow fiber membrane module to wash the hollow fiber membrane with bubbles during or after the pressurizing process.

[Claim 5]

The filtration device according to claim 4, wherein the hollow fiber membrane module has an filtrate outlet and a gas introduction port on the filtrate side, and has a raw liquid introduction port, a gas discharge port, a gas introduction port, and a raw liquid discharge port on the raw liquid side.

[Claim 6]

The filtration device according to claim 5, wherein the pressurizing process and the bubble washing process are performed by a valve provided in the hollow fiber membrane module being operated by the control device, said pressurizing process is performed by opening the gas introduction port on the

filtrate side with the gas discharge port on the raw liquid side being opened, and the bubble washing process is performed by introducing gas through the gas introduction port on the raw liquid side.

[Claim 7]

The filtration device according to claim 4, wherein said device is of an external filtration system in which the raw liquid is supplied from an outer surface side of the hollow fiber membrane, and filtrate is taken out from an inner surface side of the hollow fiber membrane.

[Claim 8]

The filtration device according to claim 4, wherein the hollow fiber membrane module includes one or more hollow fiber membrane elements mounted.

[Claim 9]

The filtration device according to claim 8, wherein the hollow fiber membrane element is of one end free type in which one end of the hollow fiber membrane is sealed without being secured.

[Claim 10]

The filtration device according to claim 4, wherein the hollow fiber membrane is made of hydrophilic polymer.

[Claim 11]

The filtration device according to claim 10, wherein the hollow fiber membrane is made of polysulfone resin hydrophilized with polyvinyl alcohol resin, polysulfone resin to which hydrophilic polymer is added, or polyvinyl alcohol resin.

[Claim 12]

The filtration device according to claim 4, further comprising an oxidation and precipitation device for ions including a reaction tank, an alkaline component storage tank, an alkaline component injection pump that injects an alkaline component into raw liquid in said reaction tank from said alkaline component storage tank, an oxidant storage tank, and an oxidant injection pump that injects an oxidant into said raw liquid from said oxidant storage tank, wherein the raw liquid from which ions are oxidized and precipitated by said oxidation and precipitation device is supplied to the hollow fiber membrane module.

[Claim 13]

The filtration device according to claim 4, further comprising a flocculating device including a storage tank for an acid component or an alkaline component for adjusting pH, an injection pump that injects the acid component or the alkaline component into the raw liquid, a flocculating agent storage tank, and a flocculating agent injection pump that injects a flocculating agent into said raw liquid from said flocculating agent storage tank, wherein the raw liquid flocculated by said flocculating device is supplied to the hollow fiber membrane module.

[Claim 14]

The filtration device according to claim 4, further comprising an oily water separator, wherein raw liquid having passed through said oily water separator is supplied to the hollow fiber membrane module.

[Claim 15]

The filtration device according to claim 14, wherein the oily water separator is an oil skimmer, a pressure floating device, a coalescer, or an electric oily water separator.

[Claim 16]

The filtration device according to claim 14, wherein the filtration device is of a circulation filtration system in which the raw liquid is circulated between the oily water separator and the hollow fiber membrane module.

[Claim 17]

The filtration device according to claim 16, further comprising a concentration tank in which the raw liquid is stored, wherein an oil content in the raw liquid stored in the concentration tank is removed by the oily water separator, then the raw liquid after removal of the oil content is supplied to the hollow fiber membrane module, and circulated liquid having passed through the hollow fiber membrane module is returned to said concentration tank.

[Claim 18]

The filtration device according to claim 4, further comprising a charge type flocculating device, wherein the raw liquid having passed through said charge type flocculating device is supplied to the hollow fiber membrane module.

[Claim 19]

The filtration device according to claim 4, further comprising a chemical liquid tank for chemical liquid washing of the hollow fiber membrane, and a chemical liquid supply pump that supplies chemical liquid stored in said chemical liquid

tank, wherein said chemical liquid is supplied to the raw liquid side of the hollow fiber membrane module.

[Claim 20]

The filtration device according to claim 4, further comprising an electrolyzed water producing device, and an electrolyzed water supply pump that supplies electrolyzed water stored in an electrolyzed water storage tank of the electrolyzed water producing device, wherein the electrolyzed water is introduced into the hollow fiber membrane module to wash the hollow fiber membrane.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

The present invention relates to a method for washing a hollow fiber membrane module and a filtration device used for the method.

[0002]

[Conventional Art]

In recent years, developments have been made in separation techniques using a hollow fiber membrane, and the techniques have been used for various purposes such as filtration of water. However, in a filtration process with the hollow fiber membrane, solid material such as suspended matter in raw liquid, which is called suspended solid (SS), adheres to a hollow fiber membrane surface or enters micropores to reduce permeation flux with time. Thus, for a long period of continuous stable filtration operation, an effective method for washing a hollow fiber



membrane as well as setting of filtration conditions is indispensable.

[0003]

Various methods for washing a hollow fiber membrane module have been studied, and can be mainly classified into a physical washing method and a chemical washing method. Various physical washing methods have been proposed including a method for forcibly scraping accretions with a sponge ball or high pressure water flow, a liquid backwash method for passing liquid such as water or permeated liquid from a filtrate side to a raw liquid side, a gas backwash method for passing pressurized gas from a filtrate side to a raw liquid side (see Japanese Patent Laid-Open No. 53-108882, National Publication of International Patent Application No. 1-500732), a bubbling method for spouting bubbles to a raw liquid side, an ultrasonic method, and an electrophoretic method. Among them, the liquid backwash method, the gas backwash method, and the bubbling method are generally widely used independently or in combination. A known chemical washing method includes a method for dissolving and removing accretions with chemical liquid such as acid, an aqueous alkaline solution, or a washing agent.

[0004]

[Problems to be Solved by the Invention]

Generally, with the conventionally known physical washing method, the washing effect is not always satisfactory, and for example, if the filtration process and the washing process are continuously performed by sequence control, permeation flux is significantly reduced within several days to several months.

Then, chemical washing is required for recovering the permeation flux. A general liquid backwash method requires providing a backwash pump, and permeated liquid obtained by membrane filtration is used for backwash, and thus the liquid backwash method is not efficient. The gas backwash method requires a large amount of high pressure gas, and thus requires a pressurized gas supply device including a large capacity air compressor, and further noise caused by a jet of high pressure gas in the backwash may become a problem. On the other hand, the chemical washing method using chemical liquid such as acid requires a process of once completely stopping filtration, then washing with chemical liquid, and removing the chemical liquid after washing. Thus, filtration must be stopped for a long period, and further, a large amount of washing waste liquid must be treated. Therefore, development of an effective physical washing method is necessary for allowing a long period of continuous filtration operation.

[0005]

The present invention has an object to provide a method for washing a hollow fiber membrane module that has a more prominent washing effect than a conventional method, and allows a long period of continuous filtration operation. The present invention has another object to provide a filtration device that is effectively used for the washing method and can reduce installation space and equipment costs.

[0006]

[Means for Solving the Problems]

The present invention provides a method for washing a hollow fiber membrane module including: performing a pressurizing process of introducing gas having pressure lower than pressure of gas discharged from a raw liquid side of the hollow fiber membrane, from a filtrate side of the hollow fiber membrane, with the raw liquid side of the hollow fiber membrane being filled with liquid; and washing the raw liquid side of the hollow fiber membrane with bubbles during or after the pressurizing process to solve the above described problems. Here, the pressure lower than the pressure of gas discharged from the raw liquid side of the hollow fiber membrane is pressure of pressurized gas when a pressurizing process with gas of a sufficiently wet hollow fiber membrane is performed, the gas passes through micropores in the hollow fiber membrane, and bubbles are released from a hollow fiber membrane surface on the opposite side, and the value of the pressure differs depending on pore diameter of the hollow fiber membrane and interface tension of liquid with which the hollow fiber membrane is wet. Specifically, the washing method of the present invention is clearly different from a conventional backwash method of a hollow fiber membrane using gas in terms of introducing gas having pressure of such a level that the gas introduced into the hollow fiber membrane is not released from an outer surface of the hollow fiber membrane. This pressure is herein referred to as a bubble point.

[0007]

The reason why the method for washing a hollow fiber membrane module of the present invention has a more prominent

washing effect than the conventional washing method is uncertain, but operations of the present invention supposed by the inventors at this moment will be described with an example of an external filtration system in Figures 4 and 5.

[0008]

Figure 4 shows flows of permeated liquid in a conventional washing method using permeated liquid, when one hollow fiber membrane is viewed in a cross section. Figure 5 shows flows of permeated liquid in introducing pressurized gas with the raw liquid side of the hollow fiber membrane being filled with liquid in the washing method of the present invention, when one hollow fiber membrane is viewed in a cross section. In the shown case, permeated liquid is passed using a pump on the filtrate side (inner surface side) of the hollow fiber membrane of one end free type in which one end is sealed without being secured. In Figures 4 and 5, arrows directed from the inside to the outside of the hollow fiber membrane show permeated liquid flowing out from a wall surface of the hollow fiber membrane to the raw liquid side outside the hollow fiber membrane, and the lengths of the arrows indicate the flow rates of the permeated liquid. As schematically shown in Figure 4, the permeated liquid introduced into the hollow fiber membrane in the conventional washing method first flows out from near a permeated liquid introduction portion with low pressure loss through the wall surface of the hollow fiber membrane to the raw liquid side, and thus permeated liquid sufficient for washing the membrane often does not flow at hollow fiber ends with high pressure loss. Thus, for uniform and effective backwash of the entire hollow fiber

membrane, a large amount of permeated liquid comparable to pure water permeability of a new membrane needs to be supplied to the hollow fiber membrane with high pressure, but such an operation is actually difficult. On the other hand, in the washing method of the present invention in Figure 5, the pressurizing process of introducing gas having pressure lower than the bubble point of the hollow fiber membrane from the filtrate side of the hollow fiber membrane is performed with the raw liquid side of the hollow fiber membrane being filled with liquid. As shown by a to d in Figure 5, as the pressurized gas causes the permeated liquid in the hollow fiber membrane to pass through the wall surface of the hollow fiber membrane and be discharged to the raw liquid side, the permeated liquid level in the hollow fiber membrane is gradually lowered. At this time, backwash is first performed near the liquid level with low pressure loss, and thus as the permeated liquid level in the hollow fiber membrane is lowered, the entire hollow fiber membrane up to the ends is uniformly washed. As described above with reference to the schematic diagram, the washing method of the present invention allows uniform washing of the entire hollow fiber membrane with the permeated liquid, and has a more prominent washing effect than the conventional washing method using permeated liquid. The above described gas backwash method is a technique of introducing pressurized gas from a filtrate side of a hollow fiber membrane to wash the hollow fiber membrane (see Japanese Patent Laid-Open No. 53-108882, National Publication of International Patent Application No. 1-500732). The technique described in the publications is to introduce pressurized gas

having pressure at a bubble point or higher of the hollow fiber membrane to cause the gas to pass to a raw liquid side of the hollow fiber membrane. In the present invention, the pressurized gas introduced to the filtrate side of the hollow fiber membrane has pressure lower than the bubble point, and as described above, the present invention is not directed to the technique of passing gas through the hollow fiber membrane to wash the hollow fiber membrane. In this point, the washing method of the present invention is different from the conventional washing method.

[0009]

According to the washing method of the present invention, as shown by e in Figure 5, after all the permeated liquid in the hollow fiber membrane is discharged from the hollow fiber membrane and washing with the permeated liquid is finished, the hollow fiber membrane is pressurized by gas from inside and forced to expand outwardly and expands as shown by the broken lines in Figure 5e. At this time, SS components adhering to the outside of the hollow fiber membrane are released or cracked, and easily removed during the pressurizing process or the washing process with bubbles after the pressurizing process. Thus, in the washing method of the present invention, the pressurizing process with gas causes a minute size change of the hollow fiber membrane, leading to release or cracks of the adhering SS components. This may be one of other reasons for providing a more prominent washing effect than the conventional washing method using permeated liquid.

[0010]

The method for washing a hollow fiber membrane module of the present invention can be performed using a filtration device including: a hollow fiber membrane module having gas introduction ports on a filtrate side and a raw liquid side, respectively; a liquid supply pump that supplies raw liquid to the hollow fiber membrane module; a pressurized gas supply device that supplies pressurized gas to the hollow fiber membrane module; and a control device that operates the liquid supply pump and the pressurized gas supply device, wherein the control device is configured to perform a pressurizing process of supplying gas having pressure lower than pressure of gas discharged from the raw liquid side of the hollow fiber membrane, from the gas introduction port on the filtrate side to the hollow fiber membrane module, with the raw liquid side of the hollow fiber membrane being filled with the raw liquid, after the supply of the raw liquid to the hollow fiber membrane module is stopped, and perform a bubble washing process of introducing gas from the gas introduction port on the raw liquid side of the hollow fiber membrane to the hollow fiber membrane module to wash the hollow fiber membrane with bubbles during or after the pressurizing process. The filtration device may include pretreatment devices such as an oxidation and precipitation device for ions according to uses as described later.

[0011]

[Embodiment]

A hollow fiber membrane used in the present invention is preferably made of hydrophilic material such as polysulfone resin hydrophilized with polyvinyl alcohol resin, polysulfone

resin to which hydrophilic polymer is added, polyvinyl alcohol resin, polyacrylonitrile resin, cellulose acetate resin, or hydrophilized polyethylene resin, because such a membrane has high hydrophilic property and thus low adhesiveness of SS components and high releasability of adhering SS components. However, a hollow fiber membrane made of other materials may be used. For example, a hollow fiber membrane made of organic polymeric material such as polyolefin, polysulfone, polyethersulfone, ethylene-vinyl alcohol copolymer, polyacrylonitrile, cellulose acetate, polyvinylidene fluoride, polyperfluoroethylene, polymethacrylic acid ester, polyester, or polyamide, or a hollow fiber membrane made of inorganic material such as ceramic may be selected according to conditions of use and desired filtration performance. The hollow fiber membrane made of polysulfone resin hydrophilized with polyvinyl alcohol resin, polysulfone resin to which hydrophilic polymer is added, or polyvinyl alcohol resin has high hydrophilic property and high heat resistance, and is thus particularly preferable. In the use of organic polymeric material, the organic polymeric material may be copolymerized with 30 mol% or less of other components, or blended with 30% by weight or less of other materials.

[0012]

In the use of organic polymeric hollow fiber membrane, a production method of the hollow fiber membrane is not limited, but any method selected from known methods can be adopted according to material characteristics and desired hollow fiber membrane performance. Generally, melt spinning, wet spinning, or



dry-wet spinning is adopted. In terms of permeability, the hollow fiber membrane preferably has an asymmetrical structure including a dense layer and a support layer. However, a hollow fiber membrane produced by melt spinning generally has a symmetrical structure, and thus the hollow fiber membrane is preferably produced by a phase inversion method such as wet spinning or dry-wet spinning.

[0013]

The pore diameter of the hollow fiber membrane used in the present invention is not limited, but preferably 0.001 to 1  $\mu\text{m}$  because of high permeability and low possibility of reduction in filtration efficiency. The pore diameter herein is a particle diameter of various reference materials such as colloidal silica, emulsion, or latex having a known particle diameter and 90% removed when filtered with the hollow fiber membrane. The pore diameter is preferably uniform. For an ultrafiltration membrane, the pore diameter cannot be measured based on the particle diameter of the reference material as described above, but molecular weight cut-off is preferably 3000 or more when the same measurement is performed using protein having a known molecular weight.

[0014]

In terms of mechanical property of the hollow fiber membrane and a membrane area as a module, the outer diameter of the hollow fiber membrane is preferably 200 to 3000  $\mu\text{m}$ , and more preferably 500 to 2000  $\mu\text{m}$ . The thickness of the hollow fiber membrane is preferably 50 to 700  $\mu\text{m}$ , and more preferably 100 to 600  $\mu\text{m}$ .

[0015]

In the present invention, the hollow fiber membrane is modularized and used for filtration. The form of the module may be selected according to filtration methods, filtration conditions, or washing methods, and the hollow fiber membrane module may include one or more hollow fiber membrane elements mounted. The form of the module includes, for example, several ten to several hundred thousand hollow fiber membranes bundled into a U shape in a module, a hollow fiber bundle having one end collectively sealed by an appropriate seal, a hollow fiber bundle having one end sealed by an appropriate seal without each fiber being secured (free state), or a hollow fiber bundle having open opposite ends. The shape of the hollow fiber membrane module is not limited, but may be, for example, cylindrical or a screen shape. In the washing method of the present invention, a "one end free" type module is preferably used in which a hollow fiber bundle has one end sealed with each fiber in a free state because a membrane surface washing effect with bubbles is extremely high.

[0016]

The filtration system with the hollow fiber membrane module used in the present invention includes external pressure all filtration, external pressure circulation filtration, internal pressure all filtration, and internal pressure circulation filtration, and can be selected according to desired processing conditions or processing performance. In terms of the membrane life, the circulation system is preferable that allows simultaneous washing of filtration coating surfaces, and in

terms of simple equipment, installation costs, and operation costs, the all filtration system is preferable. In the washing method of the present invention, the hollow fiber membranes rub against each other to provide a washing effect in washing the membrane surface with bubbles, and thus the external filtration system is preferable.

[0017]

In the present invention, the pressurizing process of introducing gas having pressure lower than the bubble point of the hollow fiber membrane from the filtrate side of the hollow fiber membrane is performed with the raw liquid side of the hollow fiber membrane being filled with liquid, and gas used for pressurization includes air or nitrogen. In the pressurizing process and the membrane surface washing with bubbles described later, the raw liquid side of the hollow fiber membrane needs to be filled with liquid. The pressure of gas used in the pressurizing process is selected so as not to exceed the lowest value among the bubble point of the hollow fiber membrane, burst pressure of the hollow fiber membrane, and durability pressure of the hollow fiber membrane module. When the bubble point and the burst pressure of the hollow fiber membrane are both higher than  $5.0 \text{ kg/cm}^2$ , the pressure of the pressurized gas is preferably  $1.0$  to  $5.0 \text{ kg/cm}^2$ , and more preferably  $1.5$  to  $3.0 \text{ kg/cm}^2$ . When the pressure of the pressurized gas is lower than  $1.0 \text{ kg/cm}^2$ , the advantage of the invention may be insufficiently provided. When at least one of the bubble point of the hollow fiber membrane, the burst pressure of the hollow fiber membrane, and the durability pressure of the hollow fiber membrane module

is lower than  $5.0 \text{ kg/cm}^2$ , the upper limit of the pressure of the pressurized gas is reduced correspondingly.

[0018]

Time for the pressurizing process with gas needs to be time or longer sufficient for completely discharging the liquid on the filtrate side of the hollow fiber membrane module, and time required for the pressurizing process differs depending on the introduction amount of pressurized gas per unit time and the volume on the filtrate side of the hollow fiber membrane module. In the external filtration system, the pressurization time needs to be set also in view of the internal volume of the hollow fiber membrane.

[0019]

In the pressurizing process with gas, backwash is performed with filtrate filled in a pipe connecting an injection portion of the pressurized gas and the hollow fiber membrane module and the hollow fiber membrane module. For example, a retaining portion such as a permeated liquid tank may be provided in a middle portion in the pipe to increase a liquid amount in backwash.

[0020]

In the present invention, during or after the pressurizing process with gas, the raw liquid side of the hollow fiber membrane is washed with bubbles, and gas used in the bubble washing process includes air or nitrogen. A supply amount of bubbles is not limited, but preferably 5 to 500 normal liter per hour (NL/h) per  $1 \text{ m}^2$  area of the hollow fiber membrane, and more preferably 10 to 300 NL/h because of high membrane washing

effect and low possibility of membrane breakage. When the "one end free" type module is used, the membrane surface washing effect with gas becomes extremely high.

[0021]

Chemical liquid washing of the hollow fiber membrane may be performed after filtration or backwash to dissolve and remove organic matter or inorganic matter adhering to the hollow fiber membrane. A method for chemical liquid washing includes a method for treatment with alkali such as an aqueous sodium hydroxide solution for removing organic or inorganic matter, a method for treatment with acid such as an acid aqueous solution for removing metal, a method for treatment with a washing agent, and a combination of the methods continuously performed, and thus the hollow fiber membrane can be reused.

[0022]

The series of processes described above including the filtration, pressurizing process with gas, washing with bubbles, and chemical liquid washing may be automatically performed by sequence control by a control device. For example, a series of washing processes including a certain period of filtration, one or more times of pressurizing process with gas and membrane surface washing with bubbles, subsequent one or more times of washing with water as required, and subsequent chemical liquid washing is automatically and continuously performed by sequence control, and the filtration and the washing process of the hollow fiber membrane and a filtration line are alternately repeated to allow a long period of continuous stable operation. Also, a so-called select switch system in which the filtration

process and the washing process are continuously repeated by the sequence control, and manual chemical liquid washing is performed when clogging becomes severe may be used for a long period of continuous stable operation.

[0023]

The method for washing a hollow fiber membrane module of the present invention provides a more prominent washing effect than the conventional washing method regardless of the material of the hollow fiber membrane and the shape of the module, and thus allows a long period of continuous stable filtration with higher permeation flux than conventional for various uses. For example, in the field of food manufacturing industry, the method can be used for sterilization, clarification, iron removal, and manganese removal of raw water; sterilization and particulate removal of washing water; sterilization and particulate removal of natural water; sterilization and purification of soy sauce; sterilization and purification of refined sake; sterilization and purification of vinegar; purification of sweet rice wine for cooling; sterilization and purification of liquid preparation; product recovery from brewing lees; sterilization, particulate removal, and purification of molasses; purification of honey; purification and concentration of enzyme and protein; purification of fermentation liquid; recovery and purification of protein from cheese whey; production of high protein milk by concentration of milk; protein recovery from fish processing effluent; concentration of fish protein; recovery of meat protein from meat processing waste; red blood cell separation from porcine blood; concentration and purification of albumin

and globulin in blood; recovery and purification of physiologically active substance from soy whey; protein recovery from soybean cooking drain; toxin removal and protein concentration of brassica protein; recovery of useful protein from potato starch industry wastewater; recovery and purification of natural color; recovery and purification of various enzymes; clarification and sterilization of liquid beverage; concentration of citrus and apple pectin liquid; and purification of fermentation liquid by recovery of bacterial cell and metabolic substance. In the medical field, the method can be used for pretreatment of producing devices of pure water and ultrapure water as raw material; pyrogen removal of washing water; production of injection water; production of dialysis water, purification of dialysate; separation, concentration, and purification of physiologically active substance such as vaccine, enzyme, virus, nucleic acid, and protein; purification of hormone; production of artificial blood; concentration and purification of polysaccharide; sterilization of hand wash water in hospitals; and sterilization of washing water for surgical tools. In the field of electronics industry, the method can be used for pretreatment of reverse osmosis membranes; final filter of ultrapure water; use point filter of ultrapure water; unit integration filter of ultrapure water; particulate removal of washing water; recovery of polishing wastewater; and recovery of dicing wastewater. In the field of chemical industry, the method can be used for concentration and recovery of coating; separation and recovery of oil solution; separation and recovery of emulsion; separation and recovery of colloid; washing and

purification of fine powder; particulate removal of washing water; purification of plating solution; and pretreatment of electrodialysis. In the field of water treatment, MLSS removal of miscellaneous water system; tertiary treatment of wastewater; recovery and reuse of wastewater; purification of wastewater by nuclear power generation; and removal of bacteria. In the field of fiber dyeing processing, the method can be used for closing of PVA desizing wastewater; recovery and reuse of fiber processing oil solution; recovery of lanolin from wool scouring wastewater; and recovery of sericin from silk processing wastewater. In the field of iron and steel processing and machining, the method can be used for recovery of barreling wastewater; recovery of buffing wastewater; treatment of rolling oil wastewater; treatment of water-soluble cutting oil wastewater; treatment of animal and vegetable oil processing wastewater; emulsion removal and washing agent recovery from degreasing wastewater; emulsion removal and recovery of rinse water; and ink removal from screen printing plate washing agent. [0024]

Next, an example of the filtration device of the present invention will be described with reference to the drawings. Figure 1 is a schematic diagram of one example of a filtration device using an external pressure type hollow fiber membrane module that can be used for performing the washing method. In the filtration device, a hollow fiber membrane module 1 in which hollow fiber membrane elements 4 are housed is partitioned by a partition plate 2 into an upper filtrate side A and a lower raw liquid side B. To the partition plate 2, protective cylinders 3



housing the hollow fiber membrane elements 4 are mounted of the same number as the number of the hollow fiber membrane elements 4. On the filtrate side A, a filtrate outlet 5 and a pressurized gas introduction port 6 are provided, and on the raw liquid side B, a raw liquid introduction port 7, a gas discharge port 8, a gas introduction port 9, and a raw liquid discharge port 10 are provided. Specifically, in the filtration device, the pressurized gas introduction port 6 is provided on the filtrate side A and the gas introduction port 9 is provided on the raw liquid side B, which is the feature of the filtration device.

[0025]

Next, an example of an operation method of the filtration device of the present invention will be described with reference to Figure 2 with an example of an external pressure all filtration system. In a state where all valves are closed, a gas discharge port valve 24, a raw liquid introduction port valve 21, and a filtrate outlet valve 23 are opened, a liquid supply pump 29 is operated to introduce raw liquid into a raw liquid side D of a filtration container 25, and after the raw liquid overflows from the gas discharge port valve 24, the gas discharge port valve 24 is closed to start filtration. Since SS components adhere to a membrane surface of a hollow fiber membrane element 26 to reduce filtration capability as a filtration time passes, the hollow fiber membrane is then washed by the method of the present invention. Specifically, after the liquid supply pump 29 is stopped, the raw liquid introduction port valve 21 and the filtrate outlet valve 23 opened in the filtration process are closed to stop filtration, then an air compressor 30 is operated

to open the gas discharge port valve 24 and the pressurized gas introduction port valve 22, and pressurized gas is introduced to the filtrate side C of the filtration container 25 to perform a pressurizing process. At this time, filtrate on the raw liquid side C and in the hollow fiber membrane is pushed out through a wall surface of the hollow fiber membrane to the raw liquid side D, and discharged to the outside by the gas discharge port valve 24. Further, simultaneously with the start of the pressurizing process or after the pressurizing process is performed for a predetermined time, a gas introduction port valve 28 is opened to perform washing with bubbles for a predetermined time. After the washing process, the gas introduction port valve 28 is closed, and the raw liquid discharge port valve 27 is opened to discharge drainage, then returning to the filtration process.

[0026]

Figure 3 shows a correlation between the processes and on/off of the operation valves for a basic operation method of the filtration device in Figure 2. In Figure 3, each circle refers to the valve being opened. Besides the basic operation method, other processes may be added as required such as a process of repeating discharge of drainage and filling with water to wash the hollow fiber membrane surface and the inside of the hollow fiber membrane module, and a flushing process.

[0027]

The filtration device of the present invention can be combined with various pretreatment devices according to uses such as: (1) an oxidation and precipitation device for ions such as iron or manganese, including a reaction tank, an alkaline

component storage tank for an aqueous sodium hydroxide, an alkaline component injection pump that injects an alkaline component into raw liquid in the reaction tank from the alkaline component storage tank, an oxidant storage tank for sodium hypochlorite, and an oxidant injection pump that injects an oxidant into the raw liquid from the oxidant storage tank; (2) a flocculating device including a storage tank for an acid component or an alkaline component for adjusting pH, an injection pump that injects the acid component or the alkaline component into the raw liquid, a flocculating agent storage tank for aluminum sulfate or polyaluminum chloride (PAC), and a flocculating agent injection pump that injects a flocculating agent into the raw liquid from the flocculating agent storage tank; (3) various oily water separators such as a filter element type oily water separator as described in pp. 52 to 54 in "Information Improvement Contributing to Promotion of Conversion to 1,1,1-Trichloroethane Alternate Washing System" (November, 1994, Industrial Structure Improvement Fund), an incorporated resin element type oily water separator, a pressure floating device, an oil skimmer, and an electric oily water separator, or a coalescer; (4) a charge type flocculating device as described in "Second CFC and Tri-ethan Alternatives Seminar Document" (November 12, 1993, Electronics Department of Nagase & Co., Ltd); or (5) an activated carbon adsorption device, and thus can remove minute suspended matter such as ions or humic materials that cannot be removed only by conventional membrane filtration, or remove an oil content that may cause clogging.

[0028]

Figure 6 shows an example of a filtration device of the present invention including an oxidation and precipitation device for ions including a reaction tank, an alkaline component storage tank, an alkaline component injection pump that injects an alkaline component into raw liquid from the alkaline component storage tank, an oxidant storage tank, and an oxidant injection pump that injects an oxidant into the raw liquid from the oxidant storage tank, wherein the raw liquid from which ions are oxidized and precipitated by the oxidation and precipitation device is supplied to the hollow fiber membrane module. A basic operation method of the filtration device will be described below. Specifically, in Figure 6, an alkaline component in an alkaline component storage tank 31 is added in a predetermined amount by an alkaline component addition pump 32 in the middle of a pipe from a raw liquid supply pump 36 to a reaction tank 35, and then an oxidant in an oxidant storage tank 33 is added in a predetermined amount by an oxidant injection pump 34. At this time, the added alkaline component includes an aqueous sodium hydroxide solution or an aqueous potassium hydroxide solution, and an addition amount is preferably such an amount that pH of the raw water is 7.8 or more, and more preferably 8.0 or more. The oxidant includes an aqueous sodium hypochlorite solution, an aqueous sodium permanganate solution, and an aqueous hydrogen peroxide solution, and the aqueous sodium hypochlorite solution is preferable because of easy handling. An addition amount of the oxidant is preferably such an amount that, when, for example, an aqueous sodium hypochlorite solution is used as an oxidant, residual chlorine concentration in filtrate having passed

through the hollow fiber membrane module is 0.1 ppm or more, and more preferably 0.3 ppm or more. In the raw liquid into which the alkaline component and the oxidant are thus injected, oxidation and precipitation reaction of ions such as iron or manganese proceeds while the raw liquid is retained in the reaction tank 35. The capacity of the reaction tank 35 is preferably such a capacity that the retention time of the raw liquid is 10 to 120 min, and more preferably 20 to 60 min. The filtration device of the present invention including the oxidation and precipitation device thus described can remove iron and manganese from groundwater that contains a large amount of ions such as iron and manganese and cannot be used as drinking water to a level suitable for drinking water, and further, the method for washing a hollow fiber membrane module of the present invention is adopted to allow a long period of stable operation.

[0029]

Next, Figure 7 shows an example of a filtration device of the present invention including a flocculating device including a storage tank for an acid component (or an alkaline component) for adjusting pH, an injection pump that injects the acid component (or the alkaline component) into raw liquid, a flocculating agent storage tank, and a flocculating agent injection pump that injects a flocculating agent into the raw liquid from the flocculating agent storage tank, wherein the raw liquid flocculated by the flocculating device is supplied to the hollow fiber membrane module. A basic operation method of the filtration device will be described below. Specifically, in

Figure 7, an acid component (or an alkaline component) in an acid or alkaline component storage tank 41 is added in a predetermined amount by an acid component (or an alkaline component) adding pump 42 in the middle of a pipe from the liquid supply pump 29 to the raw liquid introduction port valve 21, and then a flocculating agent in a flocculating agent storage tank 43 is added in a predetermined amount by a flocculating agent injection pump 44. At this time, the added acid component or alkaline component includes an aqueous hydrochloric acid solution, an aqueous sodium hydroxide solution, an aqueous potassium hydroxide solution, or an aqueous sodium carbonate solution. The flocculating agent includes an aqueous aluminum sulfate solution, an aqueous polyaluminum chloride (PAC) solution, an aqueous sodium aluminate solution, an aqueous ferric chloride solution, or a polymeric flocculating agent. As required, after the addition of the acid component (or alkaline component) and the flocculating agent, the raw liquid may be stored in the reaction tank to allow sufficient flocculating reaction, and then the liquid may be introduced into a membrane filtration device. The filtration device of the present invention including the flocculating device thus described can remove organic components from groundwater that contains a large amount of organic components called humic materials and cannot be used as drinking water to a level suitable for drinking water, and further, the filtration device can be applied to wastewater treatment containing minute suspended matter. Conventionally, addition of a large amount of flocculating agent such as aluminum sulfate or polyaluminum chloride causes severe clogging

of the hollow fiber membrane and prevents a long period of stable operation. However, the filtration device of the present invention is adopted to allow a long period of stable operation even when a flocculating agent of several hundred ppm is added.

[0030]

Figure 8 shows an example of a filtration device of the present invention including an electric oily water separator. A basic operation method of the filtration device will be described below. Specifically, raw liquid is introduced into an electric oily water separator including a charge filter 52 and a coalescer 53 by a liquid supply pump 51 to remove an oil content, and then the raw liquid is introduced into the hollow fiber membrane module 26 by the liquid supply pump 29. In the filtration device, a circulation valve 54 is partly (or fully) opened to circulate the raw liquid, and filtration is performed by a circulation filtration system. The circulated liquid is again partly or fully introduced into the electric oily water separator including the charge filter 52 and the coalescer 53 to continuously perform oily water separation and membrane filtration. A concentration tank in which the raw liquid is stored may be provided to perform concentration operation. The filtration device of the present invention including the oily water separator thus described allows application of the membrane filtration technique to uses such as separation and recovery of oil solution, separation and recovery of emulsion, treatment of rolling oil wastewater, treatment of water-soluble cutting oil wastewater, treatment of animal and vegetable oil processing wastewater, emulsion removal and washing agent

recovery from degreasing wastewater, and emulsion removal and recovery of rinse water, and further, the method for washing a hollow fiber membrane module of the present invention is adopted to allow a long period of stable operation.

[0031]

Figure 9 shows an example of a filtration device of the present invention including a charge type flocculating device, and configured to supply raw liquid having passed through the charge type flocculating device to a hollow fiber membrane module. A basic operation method of the filtration device will be described below. Specifically, the raw liquid is introduced into a charge flocculating device 61 by the liquid supply pump 29 to perform flocculation of SS components by a charge flocculating effect, and the raw liquid is introduced into the hollow fiber membrane module 26. In the charge flocculating device 61, electrodes 62 connected to a DC (or AC) power supply 63 is provided, and when the raw liquid passes through an electric field produced by the electrodes 62, electrical neutralization causes flocculation and expansion of the SS components, thereby increasing separation performance in the membrane filtration device and reducing clogging.

[0032]

Figure 10 shows an example of a filtration device of the present invention including a chemical liquid tank for chemical liquid washing of the hollow fiber membrane, and a chemical liquid supply pump, wherein the chemical liquid is supplied to the raw liquid side of the hollow fiber membrane module. A basic operation method of the filtration device will be described



below. Specifically, with the raw liquid introduction port valve 21, the gas discharge port valve 24, and a chemical liquid introduction valve 73 being opened, a chemical liquid supply pump 73 is operated to store raw liquid in a chemical liquid tank 71 for chemical liquid washing, then chemical liquid such as alkali is introduced into the hollow fiber membrane module to perform chemical liquid washing of the hollow fiber membrane module. As required, the raw liquid discharge port valve 27 and a chemical liquid discharge valve 75 may be opened to circulate the chemical liquid to perform circulation chemical liquid washing. The filtration device of the present invention including the chemical liquid washing device thus described is used to allow chemical liquid washing of the hollow fiber membrane without removing the hollow fiber membrane module.

[0033]

Figure 11 shows an example of a filtration device of the present invention including an electrolyzed water producing device and an electrolyzed water supply pump, wherein electrolyzed water is introduced into the hollow fiber membrane module to wash the hollow fiber membrane. A basic operation method of the filtration device will be described below. Specifically, electrolyte such as sodium chloride stored in an electrolyte storage tank 83 is injected into water stored in an electrolyzed water storage tank 81 by an electrolyte injection pump 84, and introduced to an electrolytic cell 86 by a liquid supply pump 85 to produce electrolyzed water. The electrolytic cell 86 includes a cell separated into an anode chamber and a cathode chamber by a diaphragm such as a porous membrane or an

ion-exchange membrane, or a cell without a diaphragm. With the raw liquid introduction port valve 21, the gas discharge port valve 24, and an electrolyzed water introduction valve 87 being opened, an electrolyzed water supply pump 82 is operated to introduce the electrolyzed water obtained and stored in the electrolyzed water storage tank 81 into the hollow fiber membrane module to wash the hollow fiber membrane module. As required, the raw liquid discharge port valve 27 and a chemical liquid discharge valve 89 may be opened to circulate the chemical liquid to perform circulation chemical liquid washing. An introduction port of the electrolyzed water may be connected to the filtrate side of the hollow fiber membrane module to perform backwash with the electrolyzed water of the hollow fiber membrane. The filtration device of the present invention including the electrolyzed water producing device and the electrolyzed water supply pump thus described is used to allow washing of the hollow fiber membrane with the electrolyzed water besides the conventional chemical liquid washing without removing the hollow fiber membrane module.

[0034]

The filtration device of the present invention may be combined into post-treatment devices according to uses such as ion-exchange resin, an ion-exchange membrane, a reverse osmosis membrane, an activated carbon adsorption device, or an activated sludge treatment device to further perform clarification and purification of the filtrate.

[0035]

Concentration liquid or drainage discharged from the filtration device of the present invention may be treated by a flocculating device including a sedimentation tank, a flocculating agent addition device, and a reaction tank, or an incinerator to reduce the amount of waste.

[0036]

[Examples]

Now, the present invention will be described in more detail with examples. The following results of the examples and comparative examples reveal that the present invention allows a long period of stable filtration.

[0037]

Example 1

Constant flow rate filtration was performed by an external pressure all filtration system under the condition of a flow rate of 560 L/h, using a hollow fiber membrane module of "one end free" type having a membrane area of 7.0 m<sup>2</sup> and including a hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol and having an average pore diameter of 0.1 μm and a bubble point of 5.0 kg/cm<sup>2</sup> or more, and using river water at 10 to 20°C as raw water. To the raw water, an aqueous sodium hypochlorite solution was continuously added so that free chlorine concentration in filtrate is 1 ppm. The hollow fiber membrane was washed once every 30 minutes by sequence control, by introducing air having pressure of 2.0 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 600 NL/h

from a lower part on a raw liquid side of the hollow fiber membrane module. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching  $1.5 \text{ kg/cm}^2$  was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 65 days.

[0038]

#### Example 2

River water was filtered in the same manner as described above except using a hollow fiber membrane made of polyvinyl alcohol resin and having an average pore diameter of  $0.1 \text{ }\mu\text{m}$  and a bubble point of  $5.0 \text{ kg/cm}^2$  or more, instead of the hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol. The filtration life evaluated by the transmembrane pressure difference was 63 days.

[0039]

#### Example 3

River water was filtered in the same manner as described above except using a hollow fiber membrane module of "opposite end secured (opposite ends of the hollow fiber membrane are secured)" type having a membrane area of  $8.0 \text{ m}^2$  and including a hollow fiber membrane made of hydrophilized polyethylene resin and having an average pore diameter of  $0.1 \text{ }\mu\text{m}$  and a bubble point of  $3.0 \text{ kg/cm}^2$  or more, instead of the hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol. The filtration life evaluated by the transmembrane pressure difference was 52 days.

[0040]

#### Example 4

Constant flow rate filtration was performed by an internal pressure circulation filtration system under the condition of a flow rate of 300 L/h, using a hollow fiber membrane module of "opposite ends secured" type having a membrane area of 5.0m<sup>2</sup> and including a hollow fiber membrane made of polyacrylonitrile resin and having a molecular weight cut-off of 3000 and a bubble point of 5.0 kg/cm<sup>2</sup> or more while raw water is passed into the hollow fiber membrane at a linear speed of 1 m/sec. The hollow fiber membrane was washed once every 30 minutes by sequence control, by introducing air having pressure of 2.0 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 500 NL/h from one end of the hollow fiber membrane. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching 1.5 kg/cm<sup>2</sup> was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 55 days. [0041]

#### Example 5

Constant flow rate filtration was performed by an external pressure circulation filtration system under the condition of a flow rate of 300 L/h, using a hollow fiber membrane module of "opposite ends secured" type having a membrane area of 5.0m<sup>2</sup> and including a hollow fiber membrane made of polyacrylonitrile resin and having a molecular weight cut-off of 5000 and a bubble point of 5.0 kg/cm<sup>2</sup> or more while raw water is passed through the

outside of the hollow fiber membrane at a linear speed of 0.5 m/sec. The hollow fiber membrane was washed once every 30 minutes by sequence control, by introducing air having pressure of 2.0 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 500 NL/h from one end of the hollow fiber membrane. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching 1.5 kg/cm<sup>2</sup> was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 49 days.

[0042]

#### Example 6

An aqueous sodium hydroxide solution was added to well water containing 2.1 ppm iron and 0.2 ppm manganese at a pH of 7.1 so as to adjust the pH to 8.2, then an aqueous sodium hypochlorite solution was added in such an amount that residual chlorine concentration was 1 ppm, and the water was further retained in 30 minutes to oxidize and precipitate iron and manganese. Constant flow rate filtration was performed by an external pressure circulation filtration system under the condition of a flow rate of 3500 L/h, using a module having a total membrane area of 49m<sup>2</sup> and housing seven hollow fiber membrane elements of "one end free" type each having a membrane area of 7.0 m<sup>2</sup> and including a hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol and having an average pore diameter of 0.1 μm and a bubble point of 5.0 kg/cm<sup>2</sup>

or more. The hollow fiber membrane was washed once every 30 minutes by sequence control, by introducing air having pressure of 3.0 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 4200 NL/h from a lower part on a raw liquid side of the hollow fiber membrane module. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching 1.5 kg/cm<sup>2</sup> was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 77 days.

[0043]

#### Example 7

Water filtration was performed in the same manner as in Example 1 except using groundwater considered to contain a large amount of humic materials and having chromaticity of 200 and potassium permanganate consumption of 45 mg/L taken in Kanto area, to which 300 ppm commercially available liquid aluminum sulfate compliant with JIS K 1450-1997 as a kind of flocculating agent was added, and which was stirred and retained for 30 minutes. The filtration life evaluated by the transmembrane pressure difference was 58 days. The obtained filtrate had chromaticity of 0 and potassium permanganate consumption of 6.5 mg/L, which is compliant with water quality standards established by Ministry of Health and Welfare Ordinance No. 69, 1992 including other items.

[0044]

#### Example 8

Constant flow rate filtration was performed by an external pressure circulation filtration system under the condition of a flow rate of 1500 L/h, using a module having a total membrane area of 49m<sup>2</sup> and housing seven hollow fiber membrane elements of "one end free" type each having a membrane area of 7.0m<sup>2</sup> and including a hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol and having an average pore diameter of 0.1 μm and a bubble point of 5.0 kg/cm<sup>2</sup> or more, and using dicing wastewater of silicon wafer for producing IC chips having SS concentration of 30 mg/L measured by a method defined by JIS K 0102 14.1 as raw water. The hollow fiber membrane was washed once every 3 hours by sequence control, by introducing air having pressure of 1.8 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 4200 NL/h from a lower part on a raw liquid side of the hollow fiber membrane module. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching 1.5 kg/cm<sup>2</sup> was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 98 days.

[0045]

#### Example 9

Constant flow rate filtration was performed by an external pressure circulation filtration system under the condition of a flow rate of 1500 L/h, using a module having a total membrane area of 21 m<sup>2</sup> and housing three hollow fiber membrane elements of



"one end free" type each having a membrane area of 7.0m<sup>2</sup> and including a hollow fiber membrane made of polysulfone resin surface-hydrophilized with polyvinyl alcohol and having an average pore diameter of 0.1 μm and a bubble point of 5.0 kg/cm<sup>2</sup> or more, and using barreling wastewater of capacitor components having SS concentration of 1000 mg/L measured by a method defined by JIS K 0102 14.1 as raw water. The hollow fiber membrane was washed once every 30 minutes by sequence control, by introducing air having pressure of 1.9 kg/cm<sup>2</sup> to a filtrate side of the hollow fiber membrane module for pressurization for 20 seconds, and then spouting air having pressure of 1.0 kg/cm<sup>2</sup> for 1 minute at a flow rate of 1800 NL/h from a lower part on a raw liquid side of the hollow fiber membrane module. During filtration operation, the transmembrane pressure difference was regularly measured, and when a filtration time up to the difference reaching 1.5 kg/cm<sup>2</sup> was regarded as the filtration life of the hollow fiber membrane module, the filtration life was 53 days.

[0046]

#### Comparative example 1

River water was filtered in the same manner as in Example 1 except that operation was stopped for 20 seconds without pressurization with gas from the filtrate side. The filtration life evaluated by the transmembrane pressure difference was 19 days.

[0047]

#### Comparative example 2

River water was filtered in the same manner as in Example 1 except that filtrate was supplied from the filtrate side by a pump for 5 seconds at a flow rate of 1120 L/h to perform backwash instead of pressurization with gas from the filtrate side. The filtration life evaluated by the transmembrane pressure difference was 23 days.

[0048]

Comparative example 4

River water was filtered in the same manner as in Example 1 except that operation was stopped for 1 minute without washing with bubbles by spouting air from the lower part on the raw liquid side of the hollow fiber membrane module. The filtration life evaluated by the transmembrane pressure difference was 20 days.

[0049]

[Advantage of the Invention]

The method for washing a hollow fiber membrane module of the present invention allows a long period of continuous stable filtration operation, and can reduce the frequency of chemical liquid washing. The filtration device of the present invention can be effectively used for the method for washing a hollow fiber membrane module of the present invention.

[Brief Description of the Drawings]

[Figure 1]

Figure 1 shows an example of an external pressure type hollow fiber membrane module used in a filtration device of the present invention.

[Figure 2]

Figure 2 shows an example of the filtration device of the present invention using the external pressure type hollow fiber membrane module.

[Figure 3]

Figure 3 shows basic operation programs of the filtration device of the present invention in Figure 2.

[Figure 4]

Figure 4 is a schematic diagram of flows of permeated liquid in conventional permeated liquid backwash.

[Figure 5]

Figure 5 is a schematic diagram of flows of permeated liquid in a pressurizing process with gas in the present invention.

[Figure 6]

Figure 6 shows an example of the filtration device of the present invention including an oxidation and precipitation device for ions.

[Figure 7]

Figure 7 is an example of the filtration device of the present invention including a flocculation reaction device.

[Figure 8]

Figure 8 shows an example of the filtration device of the present invention including an electric oily water separator.

[Figure 9]

Figure 9 shows an example of the filtration device of the present invention including a charge type flocculating device.

[Figure 10]

Figure 10 shows an example of the filtration device of the present invention including a chemical liquid tank for chemical liquid washing of the hollow fiber membrane and a chemical liquid supply pump.

[Figure 11]

Figure 11 shows an example of the filtration device of the present invention including an electrolyzed water producing device and an electrolyzed water supply pump.

[Description of Symbols]

- A: filtrate side
- B: raw liquid side
- 1: hollow fiber membrane module
- 2: partition plate
- 3: protective cylinder
- 4: hollow fiber membrane element
- 5: filtrate outlet
- 6: pressurized gas introduction port
- 7: raw liquid introduction port
- 8: gas discharge port
- 9: gas introduction port
- 10: raw liquid discharge port
- C: filtrate side
- D: raw liquid side
- 21: raw liquid introduction port valve
- 22: pressurized gas introduction port valve
- 23: filtrate outlet valve
- 24: gas discharge port valve
- 25: filtration container

26: hollow fiber membrane module  
27: raw liquid discharge port valve  
28: gas introduction port valve  
29: liquid supply pump  
30: air compressor  
31: alkaline component storage tank  
32: alkaline component injection pump  
33: oxidant storage tank  
34: oxidant injection pump  
35: reaction tank  
36: raw water supply pump  
41: acid component (or alkaline component) storage tank  
42: acid component (or alkaline component) injection pump  
43: flocculating agent storage tank  
44: flocculating agent injection pump  
51: liquid supply pump  
52: charge filter  
53: coalescer  
54: circulation valve  
61: charge type flocculating device  
62: electrode  
63: power supply  
71: chemical liquid tank for chemical liquid washing  
72: chemical liquid supply pump  
73: chemical liquid introduction valve  
74: check valve  
75: chemical liquid discharge valve  
81: electrolyzed water storage tank

- 82: electrolyzed water supply pump
- 83: electrolyte storage tank
- 84: electrolyte injection pump
- 85: liquid supply pump
- 86: electrolytic cell
- 87: electrolyzed water introduction valve
- 88: check valve
- 89: electrolyzed water discharge valve

Figure 3

- #1 Valve No.
- #2 Process
- 1 Pressurization with gas
- 2 Membrane washing with bubbles
- 3 Discharge of drainage
- 4 Filling with water
- 5 Filtration

Figure 4

- #1 Permeated liquid
- #2 Flows of permeated liquid in permeated liquid backwash with conventional pump

Figure 5

- #1 Permeated liquid
- #2 Gas
- #3 Liquid level
- #4 Flows of permeated liquid in pressurization with gas in the present invention

Figure 6

- 31 alkaline component storage tank
- 32 alkaline component injection pump
- 33 oxidant storage tank
- 34 oxidant injection pump
- 35 reaction tank
- 36 raw water supply pump

Figure 7

- 41 acid component or alkaline component storage tank
- 42 acid component or alkaline component injection pump
- 43 flocculating agent storage tank
- 44 flocculating agent injection pump

Figure 8

- 51 liquid supply pump
- 52 charge filter
- 53 coalescer
- 54 circulation valve

Figure 9

- 61 charge type flocculating device
- 62 electrode
- 63 power supply

Figure 10

- 71 chemical liquid tank for chemical liquid washing
- 72 chemical liquid supply pump
- 73 chemical liquid introduction valve
- 74 check valve
- 75 chemical liquid discharge valve

Figure 11

- 81 electrolyzed water storage tank
- 82 electrolyzed water supply pump

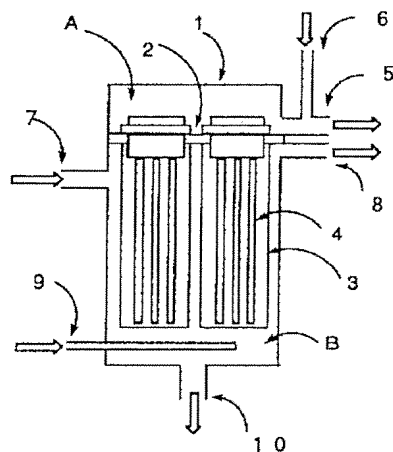


- 83 electrolyte storage tank
- 84 electrolyte injection pump
- 85 liquid supply pump
- 86 electrolytic cell
- 87 electrolyzed water introduction valve
- 88 check valve
- 89 electrolyzed water discharge valve

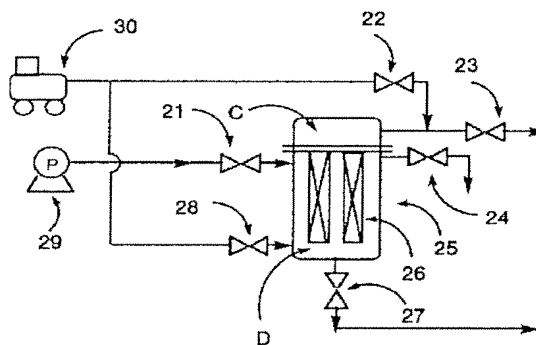
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 32 : アルカリ成分注入ポンプ  
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 36 : 原水送液ポンプ  
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 44 : 凝集剤注入ポンプ  
 51 : 送液ポンプ  
 52 : 荷電フィルター  
 53 : コアレッサー

- 54 : 循環バルブ  
 61 : 荷電式凝集装置  
 62 : 電極  
 63 : 電源  
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 72 : 薬液送液ポンプ  
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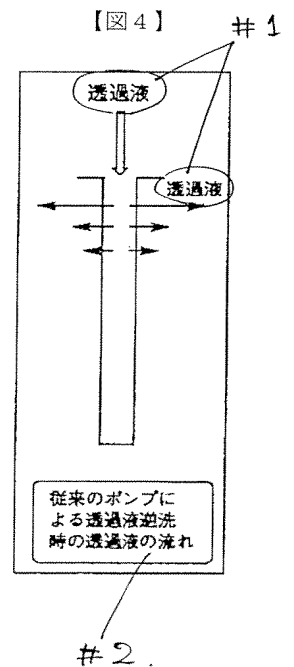
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【図2】



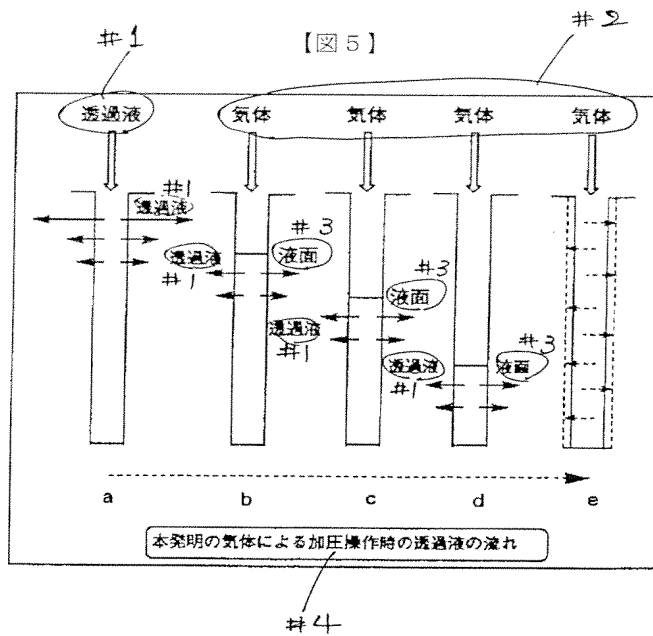
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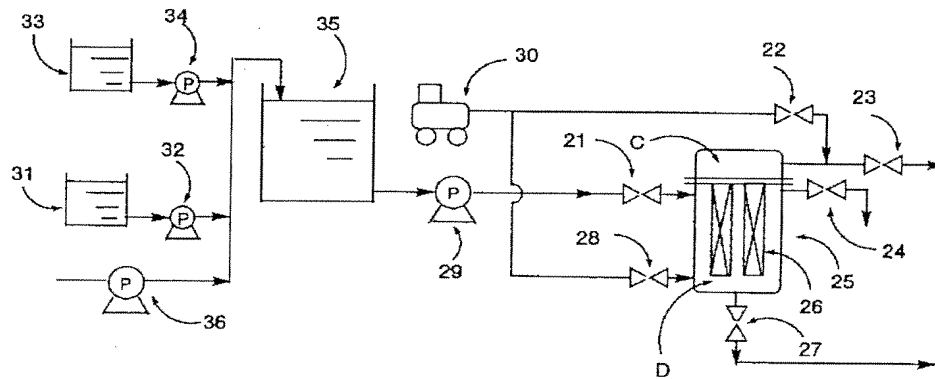
【図3】

		バルブNo.					
		21	22	23	24	27	28
工程	1	気体による加圧操作		○		○	
	2	気泡による膜洗浄				○	○
	3	ドレン排出				○	○
	4	満水	○		○	○	
	5	濾過	○		○		

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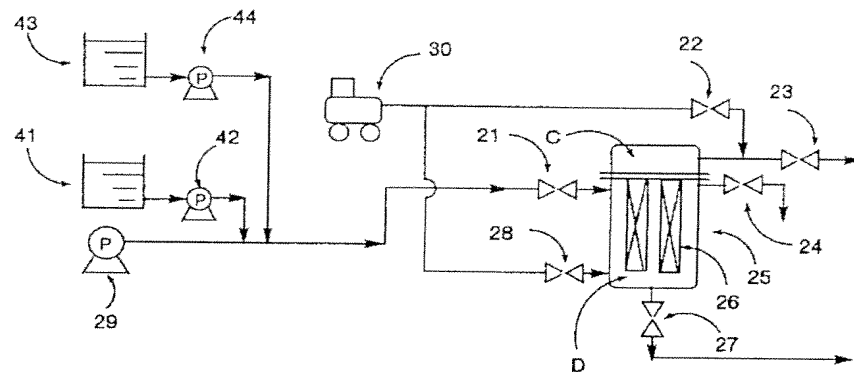


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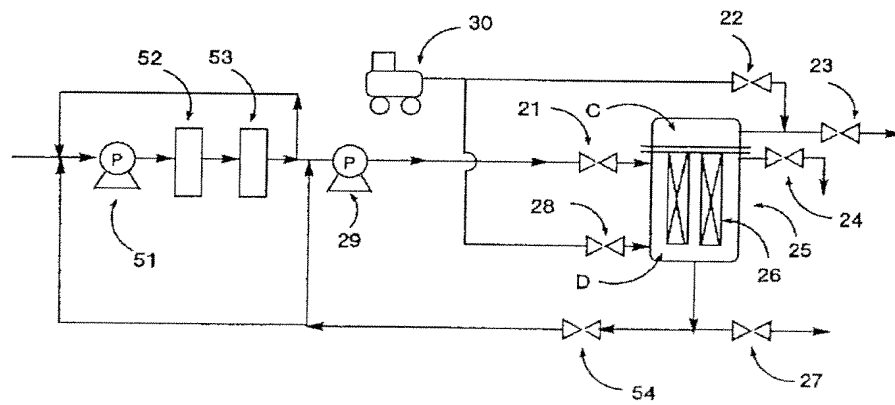
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- 32：アルカリ成分注入ポンプ
- 33：酸化剤貯留タンク
- 34：酸化剤注入ポンプ
- 35：反応槽
- 36：原水送液ポンプ

【图 7】



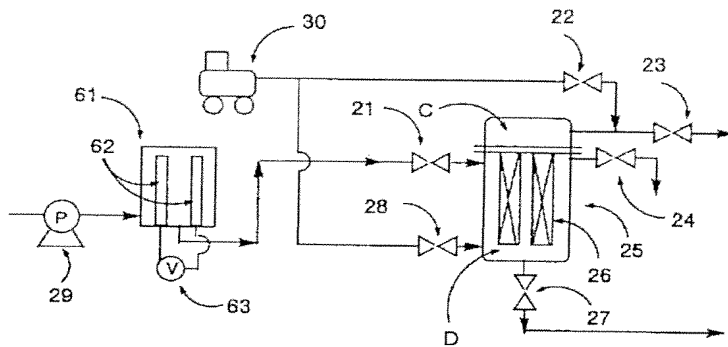
- 4 1 : 酸成分またはアルカリ成分貯留タンク  
4 2 : 酸成分またはアルカリ成分注入ポンプ  
4 3 : 凝集剤貯留タンク  
4 4 : 凝集剤注入ポンプ

【图 8】



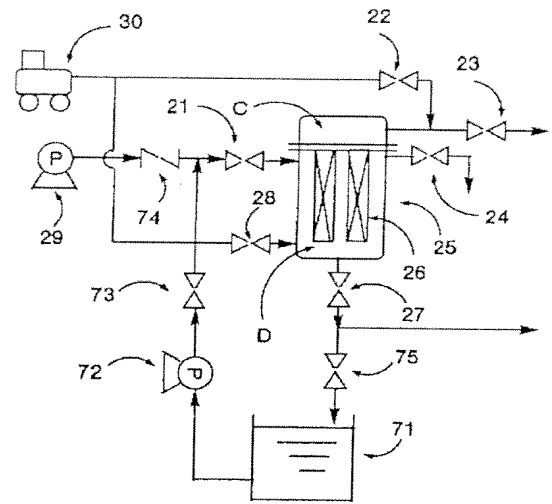
- 51:送液ポンプ  
52:荷電フィルター  
53:コアレスサー  
54:循環バルブ

【図9】



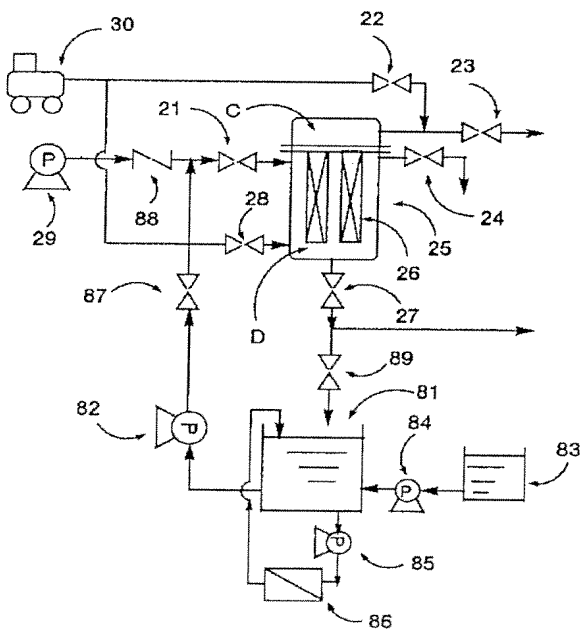
- 61：荷電凝集装置  
62：電極  
63：電源

【図10】



- 71：薬液洗浄用薬液タンク  
72：薬液送液ポンプ  
73：薬液導入バルブ  
74：逆流防止弁  
75：薬液排出バルブ

【図11】



- 81：電解水貯留タンク  
82：電解水送液ポンプ  
83：電解質貯留タンク  
84：電解質注入ポンプ  
85：送液ポンプ  
86：電解槽  
87：電解水導入バルブ  
88：逆流防止弁  
89：電解水排出バルブ